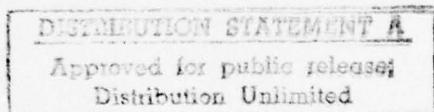
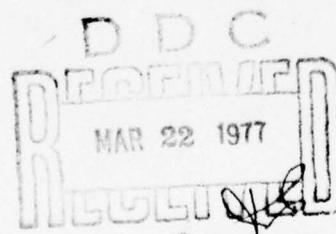


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SEMI-ANNUAL TECHNICAL REPORT

July 1, 1976

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RESEARCH & DEVELOPMENT IN THE GLASS FIBER
SODIUM-SULFUR BATTERY

Contract No. DAHC 315-73-C-0254

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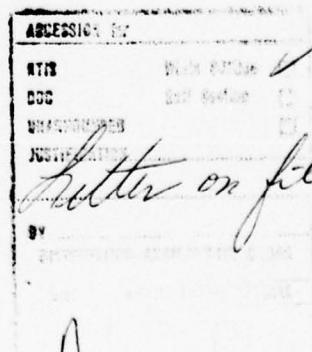
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TABLE OF CONTENTS:

	<u>Page</u>
SUMMARY	3
I. Technical Problem	3
II. General Methodology	3
III. Technical Results	3
IV. Implications for Further Research	6
INTRODUCTION	6
RESULTS	7
I. Causes of Cell Degradation	7
II. Testing of Cells and Evaluation	12
III. Back-up for Cell Fabrication and Assembly	15



SUMMARY

I. Technical Problem

In the previous reporting period, factors were identified which limited the useful life of the cell. Some of these factors were impurities in the sodium and problems with the formulation and curing of the tube sheet which, in the cell, separates the sodium and the sulfur compartments.

The technical problems which remain are to:

1. further increase the cell lifetime
2. find the best ways to mitigate the causes of cell failure
3. minimize weakening of the glass fibers at the fiber-tube sheet juncture, and
4. successfully scale up cell size to 6 A-hour and larger cells and build cells with routine success.

II. General Methodology

The problems are being attacked by building and operating various size cells and by laboratory experiments on parts of the cell assembly. For the problem of oxide removal from the sodium anode, small 1,000 fiber cells are built and operated with various treatments of the sodium. The results are then put into operation in larger 5 Amp-hour cells.

In determining the best way to use and cure the tube sheet, tensile strengths are determined on the individual fibers after they have been exposed to "tube sheet curing" conditions. More sensitive leak testing techniques were developed to ensure that tube sheets are totally fused.

The problems of scaling up to larger size cells are still being defined. The mechanical problems of assembling larger bundles mean that slightly different techniques are needed. For example, the addition of anchoring rods in the bundle roll becomes necessary as the weight of the bundle roll increases. Putting the cells into welded, sealed metal cases introduces a host of assembly problems.

III. Technical Results

During this period, cell lifetime was greatly improved. Only a few ppm of oxide species in the sodium can shorten cell operating life. We discovered how to analyze for oxide in the molten sodium by titrating with a dilute solution of barium in sodium. This

enabled the removal of all the oxide. We found, however, that the oxide content of the sodium would increase in the running cell due to migration of the water content of the component parts of the cell, e.g., glass fibers, tube sheet. A Zr/Ti alloy scavenger is now being added to the liquid sodium in the cell to pick up this latent oxide.

Even before the procedure was changed to add scavenger to the liquid sodium anode, much longer cell lives were attained. Cells of 1,000 fibers (0.5 Amp. hour) ran over 116 days on continuous charge and discharge at high rates before failure. A number of these cells ran over three months discharging to >85% of capacity at 12 times the design rate of 2 ma/cm². This gave over 1800 deep charge/discharge before failure.

Failure in these cells seemed to be due to penetration of the tube sheet by sodium. The clean sodium has extremely good wetting and spreading properties. A more thorough fusion of the tube sheet at higher temperatures for the most part solved that problem. The tube sheet made of 95-1/2% B₂O₃ - 4-1/2% Na₂O can be completely fused at 393°C, while the 94% B₂O₃, 6% Na₂O is fused at 406°C.

The 6 Amp-hour cells have shown non-variant operation using pure Na (but no scavenger) to 42 days operating at one amp-hour charge/one amp-hour discharge---mostly in the two phase region. On deep charge/discharge cycling, lifetime has been limited to about two weeks. It is believed the problem was residual oxides in the cell.

Two 40 Amp-hour cells were built during this period. One was run at 5 Amperes on 4 hour cycles and lasted two weeks. The other was cycled to about 80% depth at 3 Amps and 6 Amps, and failed in seven days -- probably from an air leak.

That the glass itself is stable to sodium, and sulfide is shown by a 14 month life on a 10 fiber bundle that was continuously C/D cycled in Na₂S₄. A single fiber assembly operated 237 days at 30 ma/cm², charging and discharging four column volumes of sodium per cycle. This gave 20,000 charge reversals and passed over 175 amp-hours per cm² of glass---the equivalent of over 10 years of coulombs passed if it were operated continuously at normal current densities.

Two of the 6 amp-hour size cells were started after being placed in completely welded sealed stainless steel cases. One lasted 16 hours and the other is still in operation after 34 days and 200 cycles of about 20% depth.

Thinner wall fibers can be used in the cell. The longest running cell reported, over 116 days, has fibers with a 6 μ wall thickness instead of the 10-15 μ thickness normally used. Some cells were built with fibers having walls of 3 μ , but they were so delicate that only one ran as long as six days.

In order to find out if the use of 316 stainless steel as a cell casing would interfere with cell operation, one 0.5 amp-hour cell had a stainless steel sheath attached to the cathode. The cell, operated at 12 times design current density, stayed normal throughout its lifetime of 75 days.

The cathode foil of 1000 Å of molybdenum on aluminum continues to work satisfactorily. A coating of 50 Å does not work. Intermediate thicknesses are being tried. A cathode foil consisting of carbon on 1145 Al alloy (no magnesium) required a "conditioning period" of seven days of cell operation before the cell resistance dropped to normal.

The tube sheet and its fusion is a critical part of the cell assembly. The paste of glass particles used in making the tube sheet must have good flow characteristics at high solids content. Studies were made to correlate particle size with good flow characteristics. Conditions were determined by which good particle size distribution could be obtained after 48 hours of grinding in a Vibra-mill.

The spherical glass particles used in the tube sheet paste are still made by dropping the particles through a $\text{CO} + \text{O}_2$ flame. Attempts to make the glass spheres various other ways, e.g., dropping the glass through a hot tube, etc., failed.

Fusion of the tube sheet causes fiber brittleness at the juncture where the fiber comes out of the tube sheet. By studying the conditions of curing, we determined that the brittleness and weakening will be minimized if the tube sheet-fiber combination is exposed to BCl_3 gas before tube sheet fusion.

The purity of the sulfur required has still not been resolved. The normal sulfur used is distilled through an 850°C column to remove organics. Cells were built that contained commercial "flowers of sulfur" that was only filtered, or sulfur that was filtered and bubbled with N_2 . The cell using only filtered (not distilled) sulfur ran in a normal fashion for 80 days. At the time of the experiment, this is as long as cells containing distilled sulfur ran. When about 0.1% lampblack was added to the distilled sulfur cathode, no bad effects were seen. The lampblack presumably contains C and H.

Improvements were made in the ability to spin the hollow glass fibers uniformly and rapidly. A new cooler was designed and built to quench the molten glass as it comes from the spinnerette. It replaces the jets of He gas used previously for quenching. The whole spinning system has been automated to allow for automatic start-up and reproducible results.

IV. Implications for Further Research

Isolating the factors limiting cell life is still the goal of the research. Although much longer lives are being achieved, cell failures do occur after about four months. This may be due to air leaks into the system, to eventual penetration of the tube sheet by sodium, or to some unknown factors. The lifetime does not seem to be coulomb or charge-discharge cycle dependent.

Scaling-up to larger sizes is presenting some mechanical handling problems. These become important when the fiber at the tube sheet is weakened. Further work on how to lessen the weakening even more is in order.

The purity of the sulfur needed for long life of the cell is still not resolved. It will probably be necessary to add various "impurities" to the sulfur catholyte to get "enhanced corrosion" for more rapid testing.

The cathode foil composition must be resolved. We are trying various thicknesses of molybdenum on 1145 alloy aluminum to find the minimum thickness that works. The original foil composition of graphite on 5005 alloy aluminum has not been ruled out.

INTRODUCTION

The sodium-sulfur battery based on hollow glass fibers as the electrolyte has been shown to have outstanding characteristics as a rechargeable power source. It should be capable of 220 watt-hour per kilogram at over 170 watts per kg. and not be damaged by thousands of deep charge-discharge cycles.

This effort is an R&D program to make this cell a practical useful reality. It is aimed at finding causes of cell failure and then finding ways to mitigate these causes. Once cell lifetime is increased substantially, as shown by a large number of deep charge-discharge cycles, 40 ampere-hour cells will be built and operated. Since the larger cells are merely a parallel extension of the smaller cells, i.e., same electrode spacing, electrode length, etc., problems in scale-up should be mostly assembly problems.

The tasks under this effort include:

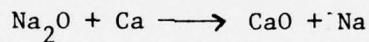
1. Back-up for fabrication and cell assembly.
2. Minimize the weakening of glass fibers at the tube sheet juncture.

3. Determine mechanisms by which harmful impurities or factors in the catholyte cause cell degradation.
4. Discover means of mitigating these harmful factors.
5. Test of cells and evaluate results.

RESULTS

I. Causes of Cell Degradation

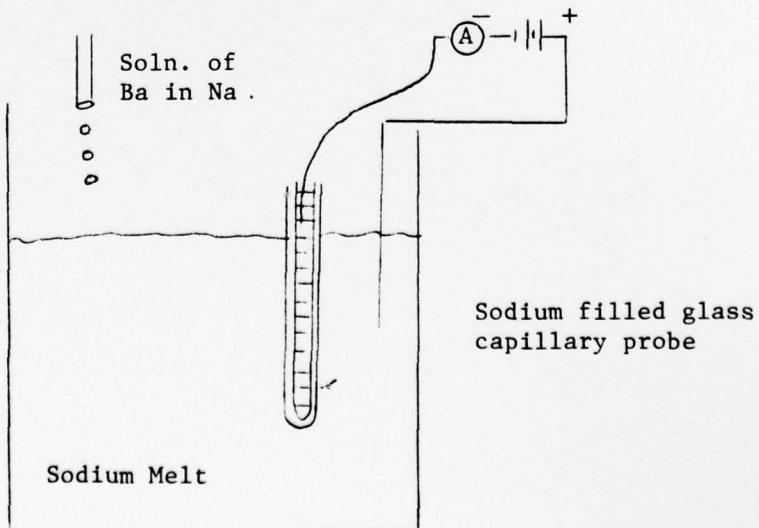
A. Removal of oxides from Na: In the previous period a method of getting rid of the oxides from the sodium was described wherein sufficient Na_2O was introduced so that upon 300°C digestion,



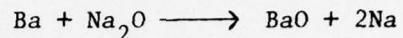
the calcium precipitated as CaO and the $\text{CaO} + \text{Na}_2\text{O}$ were filtered.

After filtration, the Na was treated at 500°C with Zr/Ti alloy to remove the final oxides to "a few" ppm or less.

A method has been developed to "titrate" the oxide present in the sodium. The experimental arrangement is as shown in figure 1.



The technique is based on the fact that when there is an excess of Ba metal dissolved in molten Na, the D.C. resistance across the glass wall of the probe has a large increase. The melt of Ba in Na is added to the pool of molten Na until the resistance rises. While there is oxide present, the barium reacts to form BaO:



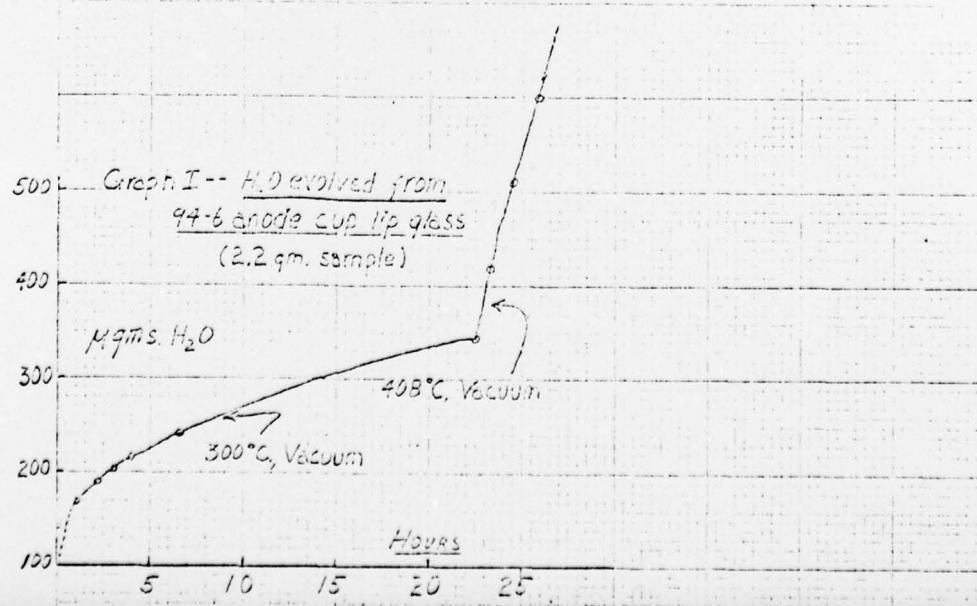
As soon as the oxide is totally removed, the current in the probe system decreases sharply.

Using the effect of Ba increasing the glass resistance, we were able to find another source of oxide in the operating cell. This source is the water content of the glass parts of the cell. Experimentally a 1,000 fiber cell was started with 15 ppm excess barium in the sodium. Initially, the cell resistances were very high. Instead of the normal resistance of 0.4 ohms, the resistance was very high and then slowly dropped. The change of resistance with time was:

14 days	100
33 days	2.6
35 days	1.1
40 days	0.5

This decrease in resistance is attributed to the barium reacting with the oxide that forms as the water migrates out of the glass structures.

Experiments were run to determine the rate at which the water migrates out of the glass. A 2.2 gram sample of the glass from the lip of the anode cup was held at 300°C under vacuum and the water enclosed was measured. Original water content was probably 0.1-0.2%. After 23 hours, the temperature was raised to 408°C. Graph I shows the results.



Attempts are now being made to keep the water content of the various glasses used in the cell as low as possible. The tube sheet glass is vacuum dried (10^{-4} mm, 300°C, 8 hours) before the final grinding. This lowers its water content to 0.09%. It is ground in a well-sealed container. Even after the tube sheet is well-fused, it can pick up water from the surroundings. In a few hours in the low humidity room (<2% R.H.) it picks up as much as 0.16% water.

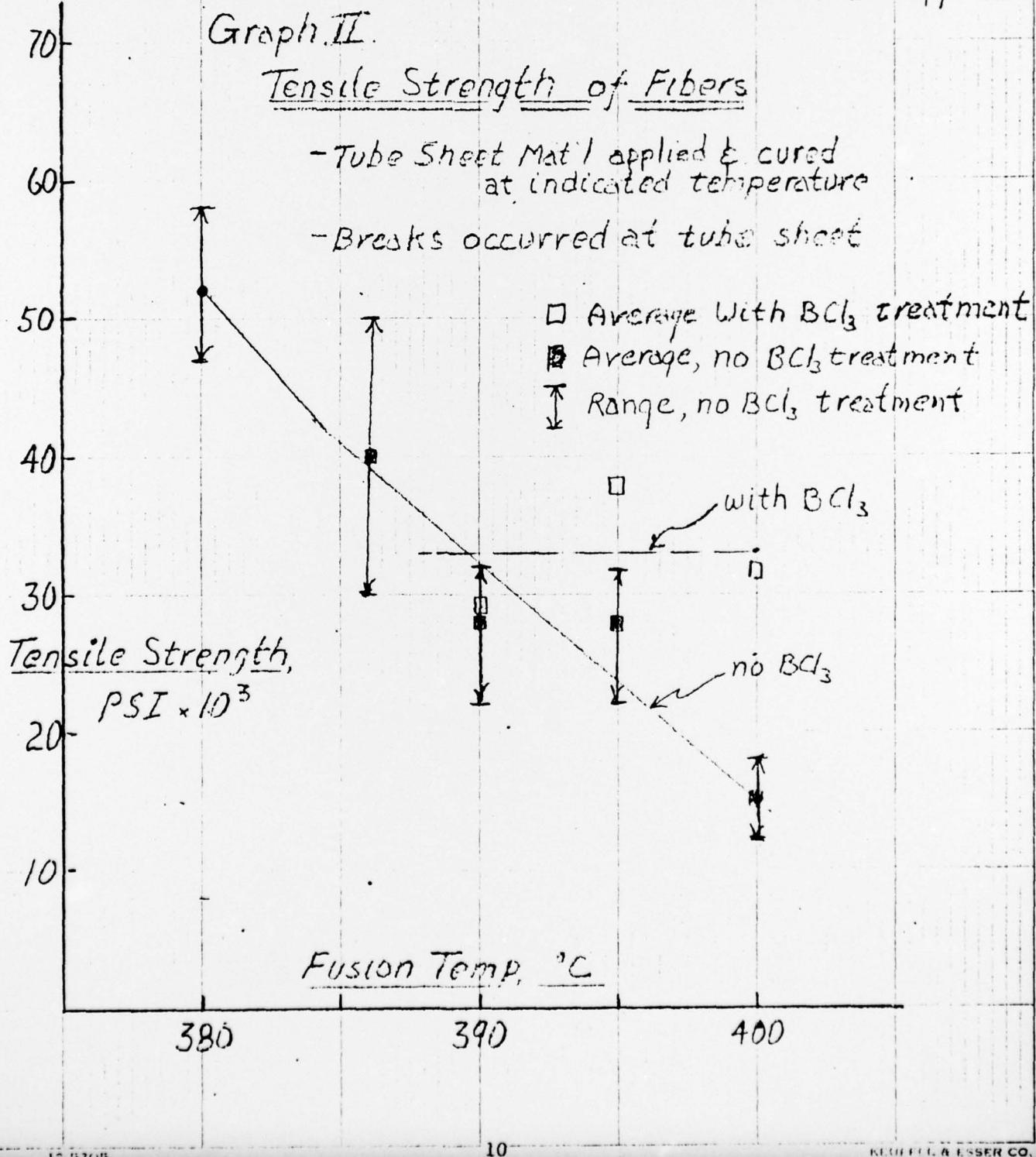
Before filling the cells, in order to dry off the interior surface moisture, some cells are now being held under vacuum at 300-330°C. To take care of the residual "bulk" moisture content of the glasses, some oxygen scavenger is now being added to the sodium in the anode cup. Scavengers soluble in the sodium, such as barium, do not work since they increase the cell resistance. An alloy of Zr/Ti is being tried as an in-situ scavenger.

B. Penetration of Sodium Through the Tube Sheet: After removing all the oxide from the sodium by the barium treatment, the sodium has greatly increased spreadability. It wets surfaces much better than if a few ppm of oxides are present. Using this "clean" sodium, cell failures began to occur which were the result of the sodium penetrating the tube sheet. The tube sheets had not been fused to solid imperious disks. Using a halogen leak detector, it was found that for a 1-1/2 hour fusion, 393°C was necessary for the fusion of 95-1/2 - 4-1/2 tube sheet material and 405°C was necessary for the fusion of 94-6 tube sheet material.

C. Fiber Weakening Under the Tube Sheet: When the temperature of tube sheet fusion is raised to completely fuse the tube sheet, the fibers at the fiber-tube sheet juncture become weaker. A study was begun to measure this effect and to try to mitigate it.

Dabs of tube sheet paste were placed on individual fibers and the fiber and tube sheet material combination was subjected to the conditions of curing and fusion. These individual fibers were then tested for tensile strength on an Instron tensile tester. The fibers almost invariably broke at the interface of the tube sheet material at the higher temperatures. Graph II shows the results for 95-1/2 B_2O_3 - 4-1/2 Na_2O tube sheet material. Complete fusion is at 393°C. It is interesting to note that at 380°C, the fibers were substantially weakened even though they broke at a location away from the tube sheet material. This work is continuing using both 95-1/2 - 4-1/2 and 96-4 tube sheet material, using 1-1/2 or 2 hour fusion treatments, and looking at the effect of BCl_3 treatment of the

•
•
•
• } No Tube Sheet
mat'l applied



tube sheet material just before fusion. Qualitatively, the BCl_3 treatment does keep the fibers from weakening drastically when the tube sheet is fused.

D. Impurities in the Sulfur: In the previous report, the effect of metal sulfides on the behavior of the cell was studied. During this period cell behavior was studied as a function of the purification treatment given to the sulfur used as catholyte. Some 1,000 fiber cells were started using commercial "flowers of sulfur" that had been (1) only melted and filtered, (2) melted, filtered and bubbled with N_2 , and (3) distilled over Vycor chips at $850^\circ C$ in the usual manner.

The results were inconclusive. Lifetimes of the cells were as follows:

<u>Sulfur Treatment</u>	<u>Cell Life</u>
Filtered only	80 days
Filtered, bubbled	15 days
Distilled	58 to over 100 days

Not enough cells with the crude sulfur were run to get a meaningful answer. It is apparent, however, that using sulfur which has been only filtered does not drastically shorten cell life.

Common impurities in sulfur are organic molecules. To simulate these, some lampblack was added to the catholyte of a 1,000 fiber cell. This should contain much adsorbed H and CH_x species. No unusual effects were observed, the cell failing by fiber failure in 44 days.

E. Corrosion of the Fibers by Sulfur-Sulfide: In order to see if gross corrosion by the catholyte is a failure mechanism within the demonstrated cell lifetimes of 80-120 days, some 1,000 fiber cells were built using fibers of 6μ and 3μ walls. The fiber outer diameters were kept at about 70μ . The fibers, therefore, were fairly delicate because of the combination of thin wall and relatively large diameter. The results are shown:

<u>Cell Fiber Size</u>	<u>Fiber Wall</u>	<u>Cell Life</u>
70 x 54	3μ	-Broke during assembly
70 x 54	3μ	-Six days
70 x 58	6μ	-Broke during assembly
70 x 58	6μ	-19 days
70 x 58	6μ	-26 days
70 x 58	6μ	-155 days

The 155 day life was the longest cell life to date - even longer than 10 μ or 15 μ walled fibers. Apparently, corrosion on the thinner wall did not shorten the cell lifetime in the lifetime range now being seen.

F. Effect of Stainless Steel Shell on Cell Lifetime: In the $\beta\text{-Al}_2\text{O}_3$ cell program at Ford Motor Company, it was found that stainless steel as a case material was unsatisfactory because it deposited electrode blocking material on the $\beta\text{-Al}_2\text{O}_3$ electrolyte. To determine whether 316 alloy stainless steel had a similar detrimental effect in our system, a stainless steel sheath was placed around the fiber bundle of a 1,000 fiber cell and electrically tied to the cathode. This cell was deep charge-discharge cycled at about 12 times design current density (24 ma/cm²). It finally failed by the mode of "fiber failure" after 75 days. The stainless steel had no apparent effects on either the lifetime or the resistance of the cell during its life.

II. Testing of Cells and Evaluation

A. Lifetimes

As various cell failure mechanisms are discovered and corrected, the lifetimes of cells assembled after that date increase. Some single fiber and some multi-fiber "devices" have been built to test certain portions or aspects of the cell construction.

1. Fiber Assemblies. To test the gross effect of Na and Na_2S_4 on the glass fiber itself, a single fiber "assembly" has been operating for over 6-1/2 months. It has been continuously discharging and charging four column volumes of sodium on each cycle at 30 ma per cm² of glass surface. It is still working after passing over 145 ampere-hours per cm² and cycling over 8000 times. This would be the equivalent in coulombs passed to over eight years of constant operation at normal current densities. These results show that the glass itself is not harmed by the passage of current or the cycling of current.

Another 10 fiber dianode assembly operated for 14 months in Na_2S_4 . The glass apparently is not corroded in this catholyte.

2. Cells of 1000 Fibers (0.5 ampere-hour cells). These cells are the workhorses for determining the effects of changing various conditions of cell assembly.

The longest lived cell operating on deep charge-discharge cycling is now over 96 days. It is discharging to over 80% depth on each cycle. This cell is discharging in 1/2 hour and charging in 1/2 hour. It operated at about 12 times the design current density of 2 ma/cm². It has an unknown amount of excess Zr/Ti scavenger in the anode chamber. This cell has undergone over 2300 cycles and passed coulombs equivalent to over three years.

Another 1,000 fiber cell charge-discharge cycling at 12% depth, is still operating after 116 days. This cell has fibers with 6μ walls. It was deep charge-discharged for 37 days when the anode lead lost contact with the sodium in most of the fibers. It has been operated since then at normal rates, but one hour cycles.

Other 1000 fiber cells are operating on shallow cycles, but at high rates. One cell is operated to only 5% depth on four minute cycles at about 15 times design current density. So far, it has gone over 95 days making over 33,000 cycles.

These cells seem to fail in one of two modes. The first is fiber failure, with a few fibers breaking and then more fibers breaking. This is seen by a decrease in the open circuit and terminal voltages. Cell resistance also decreases. The cell then discharges itself. The other failure mode happens within a few minutes -- or seconds -- after the cell has been switched from discharge to charge. There occurs a rather massive shorting. It is tempting to speculate that fiber failure here is due to deposition of sodium metal in a crack formed inside the fiber from corrosion by oxide.

3. Cells of 3,000 Fibers (6 Ampere-hours). These cells are scaled up from the 0.5 Ampere-hour cells, but built using the same techniques that larger cells would use. In the past, one of these cells ran as long as 78 days on shallow charge-discharge at low current density.

The longest lived of these 6 A-hour cells to date cycling to 80% depth is 16 days. It had 190 deep cycles. Failure was due to an instrument failure in the charge-discharge circuit. It contained clean Na with an unknown amount of excess scavenger. These larger cells do not last as long as the smaller cells. It is not known whether the problem is due to the extra strains put on the cells during assembly because of their size, or because the larger number of fibers gives a statistical chance of earlier failure.

One of these 6 A-hour cells ran 42 days on continuous four hour cycles at 0.5 amperes (16% depth). The catholyte was mostly in the two phase region. Although the cathode foil in this cell was carbon on 5005 aluminum, the cell showed no resistance increase during its life. A similar cell had no resistance increase during its 27 day life.

Two 6 ampere-hour cells were sealed in stainless steel cases and operated. This required development of different handling and welding techniques. One cell only ran 16 hours before failure, but the other one is still operating after 36 days and over 200 shallow cycles. This cell is cycling at 2 hour charge and 2 hour discharge to 20% depth. It is a completely sealed cell.

4. Cells of 15,000 Fibers (40 Ampere-hour). Several of these larger cells were built and operated during this period. Lifetimes were relatively short. The first operated one week and failed - probably due to air leaking into the cell and destroying the fibers. It was cycled to about 80% depth but at rates of 6 to 10 hours.

The other 40 Ampere-hour cell was operated on four hour cycles at 5 amperes (25% depth). It failed after two weeks.

These cells failed drastically at the tube sheet releasing molten sodium into the sulfur catholyte. The heat released caused the glass case of the cell to break. A pressure release valve will be placed on the next large cells built.

Again, the cause of the shorter life for the large cells may be due to handling and assembly problems or may be due to the statistical chance of having more fibers.

B. Effect of Cathode Foils

In the last report, the effect of aluminum cathode foils containing magnesium was shown. Resistance of operating cells was shown to increase if magnesium is present. This has not been seen with the 6 ampere-hour cells, although less total coulombs per cm has been passed in these larger cells.

Cathode foils of 1000 Å of molybdenum on 1145 alloy aluminum are being used to take the place of the carbon coated 5005 (Mg containing) aluminum. Thinner coatings of molybdenum are being tried. A 50 Å coating is not enough. The cell made with this foil has a very high resistance. Intermediate thicknesses are being tried.

A foil of carbon on 1145 alloy aluminum was tried in a cell. It started out with very high resistance. However, the cell resistance dropped to almost normal after seven days of cell operation. The cell failed in 33 days of shallow cycling. A cell with no carbon on 1145 aluminum failed in 32 days without a lowering of its initial high resistance.

III. Back-up for Cell Fabrication and Assembly

A. Fiber Spinning

A number of improvements were made in the fiber spinning apparatus to give better and more uniform fibers. The most important was the development of a new type cooler to quench the molten glass as it emerges from the spinnerette. Instead of using jets of air to quench the molten glass, the glass drops through a water cooled block that is machined to give a close fit around the glass thread. This gives a cooling that is reproducible and does not cause vibrations in the glass fiber (as from the air jets). Since cooling is the limiting factor on fiber spinning rate, better cooling allows faster spinning of fibers.

Another major improvement was the completion of the automatic sequence timer that automatically prepares a glass cullet charge for spinning. The upper section of the glass melt tank is first heated to 890°C to lower the glass viscosity. The lower section of the tank is then heated until a drop of glass falls from the spinnerette. Air is blown on the spinnerette to cool the glass and seal the bottom of the tank. A vacuum is applied above the glass to remove bubbles in the melt. The vacuum is increased slowly over eight minutes to gently debubble the melt without foaming. After two hours the vacuum is released and the upper tank temperature dropped to normal spinning temperature. Since these operations take place in the night, the spinning operation can begin with the start of the day.

Other improvements in the fiber spinner include new softer lay-down belts for moving the glass fibers, new constant flow glue applicators, and new plug-in hot N₂ fiber glass cutters. The last item allows a quick replacement when a cutter burns out instead of wasting one-half of a day's run.

As a consequence of the above improvements, most of the fiber glass pulled is of excellent quality with very few breaks and almost no double seals.

B. Tube Sheet Development

Vibra-mill grinding of the glass for the tube sheet material gave excellent results. Vibra-milling was done for up to 100 hours in the presence of 2% hexadecylamine at 90°C. Forty-eight hours gave material that was marginally good. The mill must be sealed to keep water out since the glass is so hygroscopic. Figure 2 shows the effect of particle size distribution on "goodness" of the tube sheet material. The powder from the first Sweco Vibra-mill run did not work. The rate at which the glass

Fig. 2
Summary Mass Population
Curves for Eight
Samples

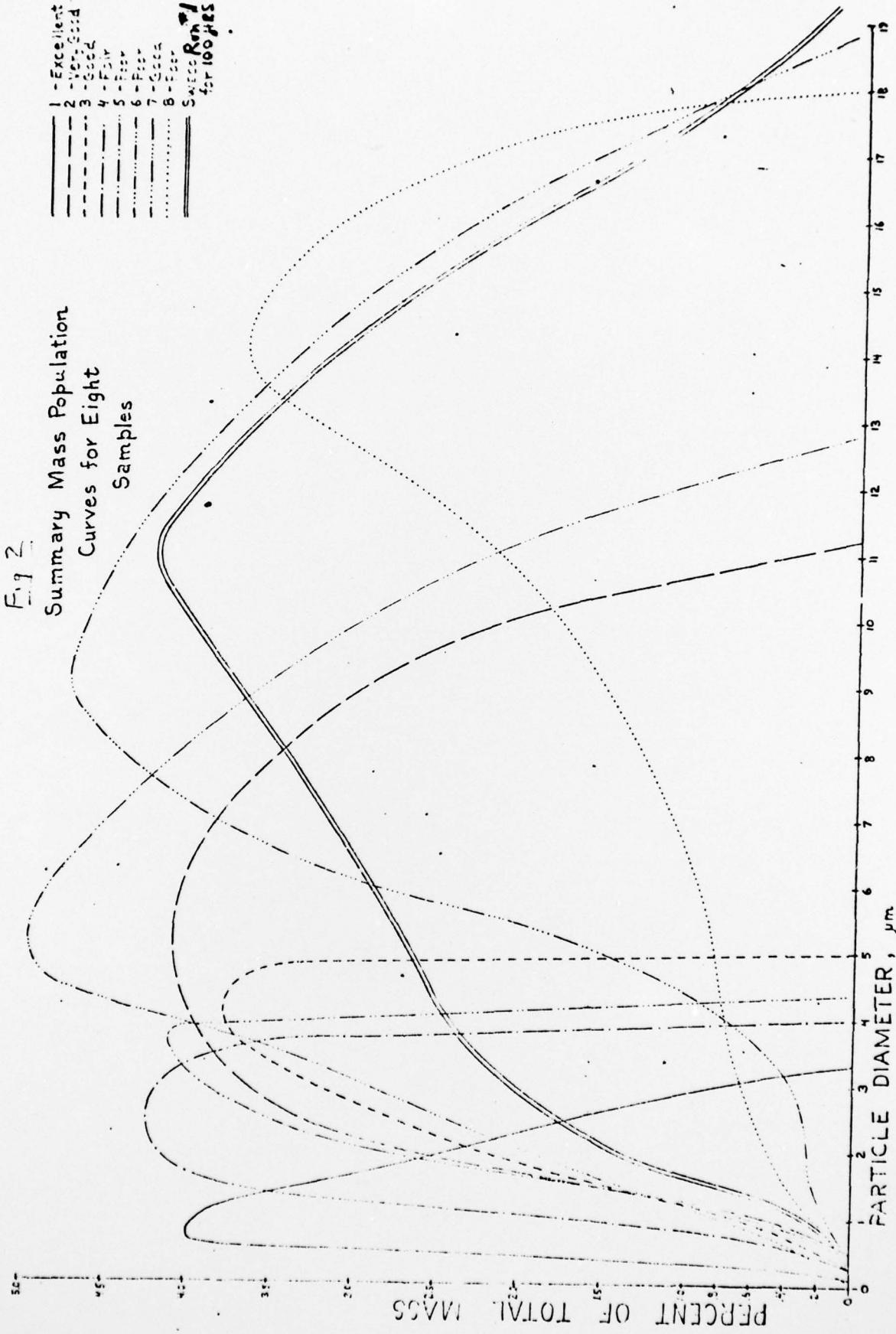


Fig. 3

SWECO RUN #1 (DM3-C mill, half-full)

CORNING 7123 GLASS (95.5-4.5)

3.9 Kg of -16 mesh glass + 72 Kg grinding media (1/2 inch Al_2O_3 cylinders)

95 \pm 5°C

1% and 1½% Hexadecyl Amine as indicated

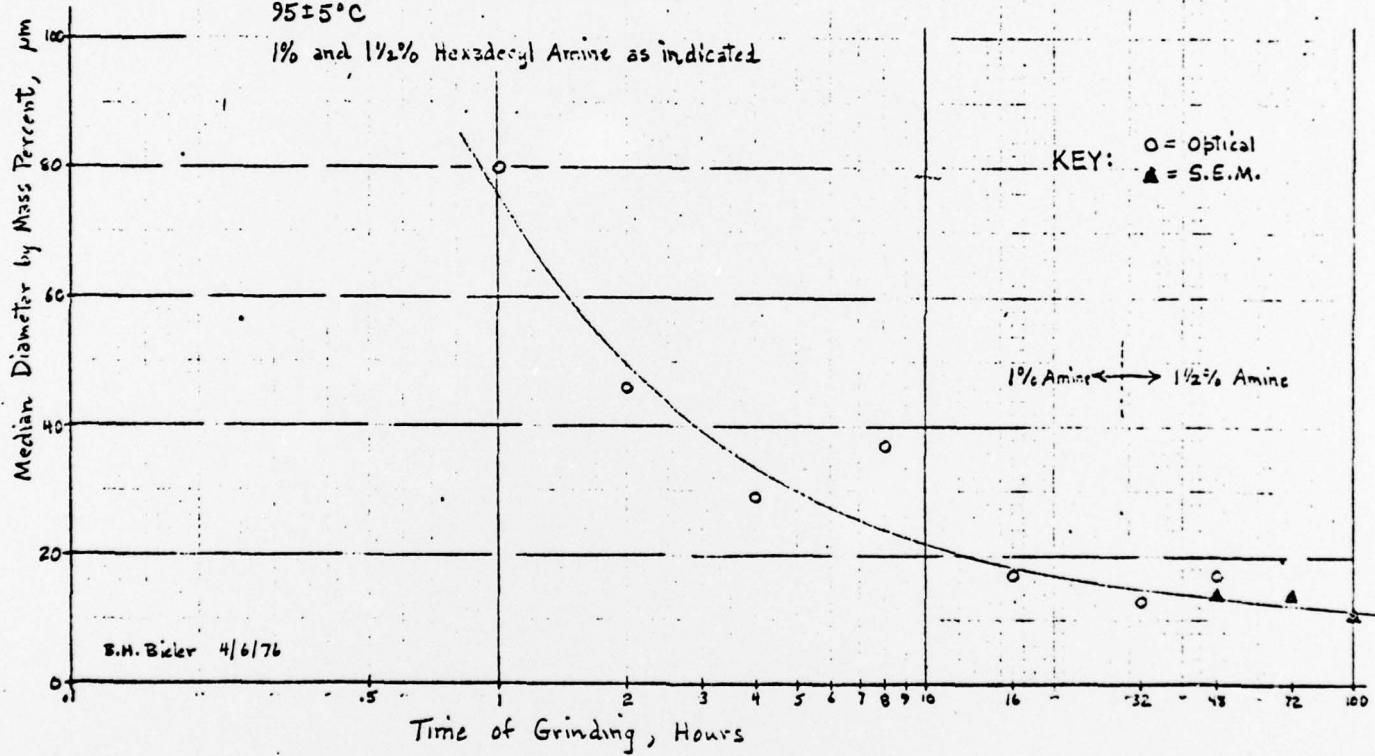


Fig. 4. GLASS PARTICLE SIZE DISTRIBUTIONS

SWECO RUN #1 (Dx3C mill, half-full)
CORNING 7123 GLASS (55.5-45)

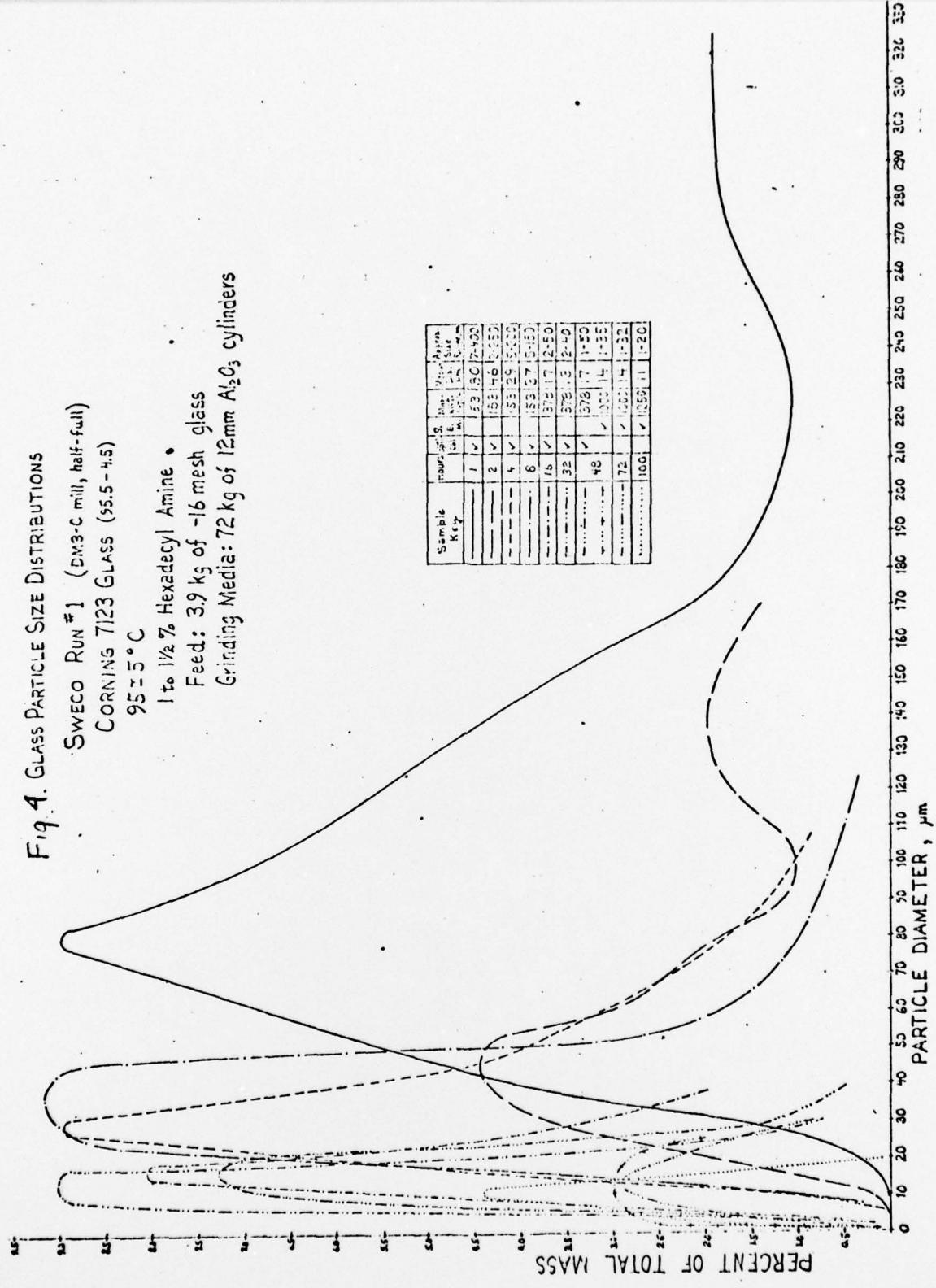
95±5°C

1 to 1½% Hexadecyl Amine

Feed: 3.9 kg of -16 mesh glass

Grinding Media: 72 kg of 12mm Al_2O_3 cylinders

Sample K.T.	Initial Size in. (mm)	Final Size in. (mm)	Volume Change
1	✓	1.53 (3.9)	-7.52
2	✓	1.53 (3.9)	-7.52
3	✓	1.53 (3.9)	-7.52
4	✓	1.53 (3.9)	-7.52
5	✓	1.53 (3.9)	-7.52
6	✓	1.53 (3.9)	-7.52
7	✓	1.53 (3.9)	-7.52
8	✓	1.53 (3.9)	-7.52
9	✓	1.53 (3.9)	-7.52
10	✓	1.53 (3.9)	-7.52
11	✓	1.53 (3.9)	-7.52
12	✓	1.53 (3.9)	-7.52
13	✓	1.53 (3.9)	-7.52
14	✓	1.53 (3.9)	-7.52
15	✓	1.53 (3.9)	-7.52
16	✓	1.53 (3.9)	-7.52
17	✓	1.53 (3.9)	-7.52
18	✓	1.53 (3.9)	-7.52
19	✓	1.53 (3.9)	-7.52
20	✓	1.53 (3.9)	-7.52
21	✓	1.53 (3.9)	-7.52
22	✓	1.53 (3.9)	-7.52
23	✓	1.53 (3.9)	-7.52
24	✓	1.53 (3.9)	-7.52
25	✓	1.53 (3.9)	-7.52
26	✓	1.53 (3.9)	-7.52
27	✓	1.53 (3.9)	-7.52
28	✓	1.53 (3.9)	-7.52
29	✓	1.53 (3.9)	-7.52
30	✓	1.53 (3.9)	-7.52
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32	✓	1.53 (3.9)	-7.52
33	✓	1.53 (3.9)	-7.52
34	✓	1.53 (3.9)	-7.52
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89	✓	1.53 (3.9)	-7.52
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93	✓	1.53 (3.9)	-7.52
94	✓	1.53 (3.9)	-7.52
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97	✓	1.53 (3.9)	-7.52
98	✓	1.53 (3.9)	-7.52
99	✓	1.53 (3.9)	-7.52
100	✓	1.53 (3.9)	-7.52



was ground in this run is shown in Figure 3 and the particle size distribution is shown in Figure 4. It is apparent that there is not a great deal of size reduction after 48 hours. This may be due to a poor seal on the Sweco Vibra-mill on this run allowing the entrance of moisture.

On the next run, where great care was taken to keep everything dry, excellent tube sheet material was obtained in 48 hours of milling. The milling was done at 80-90°C with 3/4% hexadecylamine and 3/4% dodecyl amine as grinding aids.

It is very difficult to sieve the finely ground dry glass that goes into the tube sheet paste. When it is dry it has a very high static charge. The sieving technique now used is to let it pick-up enough room moisture to lose its static charge (a few minutes or less), and, after sieving, dry it at 275-300°C, 10^{-4} mm, for at least 8 hours. This drops the water content back down to 0.09% or less.

The hot tube technique for spheroidizing glass particles to be used in the tube sheet mix has not worked. It was hoped that this would replace the technique of making dry glass spheres wherein the glass is dropped through a flame of CO and O₂. When the glass particles are dropped into the hot tube in order to melt and spheroidize, they tend to stick to the wall. A graphite wall caused less sticking at first, but then lost its effectiveness.

There were brief attempts at making tube sheets other than by the method now in use of extruding a paste while the cell is being rolled and assembled. In these methods generally the fibers were pushed or vibrated into a pack or disk of the tube sheet material. In all cases there was a poor distribution of the tube sheet material around the fibers.

C. Assembly of Cells

Few changes have been made in its actual assembly of cells, other than those cells which are placed in the all-steel cases. With the good wetting characteristics of the oxide-free sodium, no BC₁₃ treatment is needed on the cell before filling with sodium. All the 50 μ I.D. fibers fill under one atm of pressure.

In order to get more physical rigidity in the larger tube bundles, some "anchoring" rods are now being used to tie the tube sheet to the bottom of the bundle. These 60 μ glass rods are rolled in at the bottom spacer tape and extend up into the tube sheet. They help hold the fiber-foil bundle more rigid with respect to the tube sheet while the bundle is handled preparatory to its being filled.

The assembly procedure for the cells put into metal cases is a whole different story. These procedures are now being worked out. Welding creates expansions and strains on the fiber bundles which must be allowed for.

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